Journal of Applied Sciences Research, 8(3): 1646-1653, 2012
ISSN 1819-544X
This is a refereed journal and all articles are professionally screened and reviewed

ORIGINAL ARTICLES

Influence of Magnesium and Stirrer model in Production of Al–fly ash Composites – A Taguchi Approach

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ABSTRACT

Aluminium Matrix Composites (AMCs) has provided the potential applications in aerospace and automotive industries owing to their superior strength to weight ratio. Commercial Al and fly ash particles are chosen as matrix and reinforcement material respectively to produce composites by stir casting process. Achieving homogeneous distribution of reinforcement within the matrix is one such challenge in the stir casting process, which affects directly on the properties and quality of composites. In this research, an attempt has been made to study the influence of weight percentage of fly ash, wetting agent magnesium and the impeller model on the mechanical properties and achieving the effective flow pattern to uniformly disperse the fly ash particles in the molten Al matrix through modified two step stir casting method. Optimum parameters were identified for attaining the maximum mechanical properties such as hardness and tensile strength of composites by the application of Taguchi method, Analysis of Variance (ANOVA) and validated the results by confirmation test. The microstructure of the produced composites was examined by scanning electron microscope.

Key words: Aluminium Matrix Composites; fly ash; Modified two step stir casting, Taguchi

Introduction

Aluminium Matrix Composites (AMCs) have attracted more attention due to their combined properties such as high specific strength, high stiffness, low thermal expansion coefficient and superior dimensional stability at elevated temperatures as compared to the monolithic materials. In this study, fly ash particles which are extracted from residues generated in the combustion of coal were chosen as reinforcement material.

Among various processing techniques, stir casting appears to be most promising route for production of aluminium matrix composites because of simplicity and able to produce the composites in large scale economically. Since the stir casting allows a conventional metal processing route to be used, it minimizes the final cost of the product. It is observed that as volume percentage (3–10%) of fly ash increases, hardness, tensile strength and elastic modulus increases in Al–fly ash composites (Rohatgi and Guo, 1997).

Inherent difficulties associated with stir casting method are non-wettability of the reinforcement particles by liquid Al, segregation of particles, higher porosity and extensive interfacial reaction due to higher processing temperature. Porosity in stir castings is produced as a result of gases entrapped in melting and during stirring/mixing, which form gas bubbles, causes large porosity (Hashim et al., 1999, 2003). The degree of porosity depends on the processing parameters, type of matrix and reinforcement, weight fraction of reinforcement and interfacial reaction. The distribution of the reinforcement particles in the molten matrix depends on the geometry of the mechanical stirrer, stirring parameters, placement of the mechanical stirrer in the melt, melting temperature, and the characteristics of the particles added (Girot et al., 1987).An uneven distribution of reinforcements could lead slip of dislocations and initiation of micro cracks which causes premature failures in the composite under the application of load. Agglomeration of reinforcement would cause brittle nature in the composites and the formation weak bonds lead to the reduced mechanical properties.

Wettability can be defined as the ability of a liquid to spread on a solid surface, and represents the extent of intimate contact between a liquid and a solid. Though the fly ash particles have a density of approx. 2.7 g/cm^3 (densities of major constituents of fly ash are SiO_2 - 2.65 g/cm^3 and Al_2O_3 -3.5 g/cm^3) than the commercial Al 2.2 g/cm^3, when they were incorporated into the molten Al, they were observed to be floating on the molten Al surface due to the high surface tension .Moreover the liquid Al is readily covered with an oxide layer during the fabrication process below 1000K which leads poor wettability .

Wettability can be improved by increasing the surface energies of the solids, decreasing the surface tension of the liquid Al matrix and decreasing the solid / liquid interfacial energy at the reinforcement matrix interface. Magnesium can be used as wetting agent during the composite synthesis and it scavenges of the oxygen from

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the reinforcement surfaces, thus thinning the gas layer and improving wetting action with the Al matrix. The degree of stirring depends on various factors, such as the type and shape of the impeller, and its location relative to the molten surface as well as the wall of the crucible, stirring speed and time.

According to the previous study, it was found that 15 minutes stirring time with 300 rpm stirring speed showed good result in micro structure and mechanical properties as well. Hence in this study, impeller model was considered as process parameter by keeping stirring time and speed as constants. In order to achieve a good homogeneous distribution of the fly ash particles in Al matrix, the impeller must be designed such that it creates vortex in the composite slurry. Hence, the proper understanding of the impeller parameters is essential. Various investigators (Surappa and Rohatgi, 1978) have used both axial and radial flow impellers for the synthesis of MMC. When the fly ash content in the melt increases, the inter particle distance is lowered and forms clusters which leads to the formation of gas layers between the particles. These layers drag the fly ash particles together leading to spontaneous rejection from the Al melt. In order to distribute the fly ash lumps into small aggregates, it is important to increase the shear force in the composite slurry. Radial impellers produce more shear (relative motion between different layers of fluid), thereby shearing away the particle coming together. So that it is necessary to systematic study the influence of process parameters such as impeller model, weight percentage of fly ash and magnesium on the distribution of fly ash particles in molten Al and mechanical properties of composites which are fabricated by modified two step stir casting method. The prime objective of this study is to use Taguchi method for predicting the optimum process parameters that give the highest hardness and tensile strength to the composites.

Experimental Work:

In this study, 99.5% pure aluminium ingot was used as the matrix material and fly ash particles with average size of (50-100μm) was used as the reinforcement which consists of SiO_2 -54.27%,Al_2O_3-34.73%, Fe_2O_3 - 6.1%, CaO -2.4%,MgO-2.1%.

Fig. 1: Stir casting setup.

In this research, Al - fly ash composites were produced by modified two step stir casting. Schematic of stir casting setup is shown in Fig.1.Al was charged in to the graphite crucible, and the furnace temperature was raised up to liquidus temperature 670˚C in order to melt the Al scraps completely and then the stirring was started at 300 rpm. During stirring, magnesium and then preheated fly ash particles were added into the crucible at the side of the vortex. Stirring was carried out for 5 minutes.

Then the melt temperature was dropped to 620˚C to obtain the semi- solid state. Stirring was done for 5 minutes in the semi-solid state. Then the composite slurry was again reheated to the liquidus temperature of 655˚C and stirred at 300 rpm for 5 minutes. During stirring process, the impeller was frequently moved vertically within the slurry at the rate of 2mm/s by means of stirrer position control unit during stirring process. Rotary as well as reciprocating movement of impeller in the composite slurry during stirring was done through a sliding mechanism .Finally the composite slurry was poured into the steel mould to solidify. It was degassed by purging hexachloroethane tablets and Argon gas was blown at the rate of 2CC/min in to the furnace during the process to minimize the high temperature oxidation problems of aluminium and magnesium. The melting was done in an electrical resistive furnace (2Kw-1Kg capacity).Temperatures were measured with a thermocouple (+/-3 K error).

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Mechanical properties:

Hardness test was performed on composite specimens. The hardness values of the specimen were measured using Brinell hardness testing system with 10mm diameter at a load of 500 kg. The loading time was 30 seconds. Three readings were taken on each specimen to eliminate possibility of segregation and mean value was taken as the hardness of the composites. Tensile strength tests were carried out on composites using a computerized UTM testing machine as per the ASTM E-8 standards. Three samples were tested for each composition and mean value was taken as the tensile strength.

Results:

Design of Experiments (DOE):

Taguchi's parameter design provides a systematic and efficient methodology for determining optimum parameters which have an effect on the process and performance. It eliminates the need for repeated experiments and thus saves time, material and cost. By studying the effect of individual factors on the results, the combination of optimum parameters can be determined. In the Taguchi method, the term 'signal' expresses the desirable value (mean) and the term 'noise' expresses the undesirable value (standard deviation) for the output quality characteristics. In the present work, "Larger is better" S/N ratio is used to predict the optimum parameters because a higher hardness and tensile strength of the composites was desirable. In the present investigation, hardness and tensile strength tests were conducted in the composite material as per the L9 orthogonal array. Accordingly, 9 experiments were carried out and each experiment was repeated thrice in order to minimize the experimental errors. The factors and the corresponding levels which have been used are presented in Table 1.

Table 1: Factors and levels.

Level	Fly ash wt.% (A)	Mg wt.% (B)	Impeller model (C)
I	10	0.5	Radial flow -Rushton type (1)
II	15	1.5	Radial flow (2)
III	20	4	Axial flow - 45˚ (3)

Table 2: S/N ratios and measured values for hardness and tensile strength of composites.

Exp .No	Fly ash wt.% (A)	Mg wt.% (B)	Impeller model (C)	Signal/Noise Ratio		Measured Values	
				Hardness	Tensile strength	Hardness(BHN)	Tensile strength (MPa)
1	10	0.5	1	30.629	40.256	34	103
2	10	1.5	2	33.255	40.984	46	112
3	10	4	3	30.881	40.000	35	100
4	15	0.5	2	33.442	41.289	47	116
5	15	1.5	3	34.320	41.583	52	120
6	15	4	1	32.465	40.906	42	111
7	20	0.5	3	33.624	40.340	48	104
8	20	1.5	1	34.807	40.984	55	112
9	20	4	2	34.151	40.506	51	106

Table 3: ANOVA analysis for Hardness and Tensile strength.

Factors	Dof	Hardness			Tensile strength		
		F	P value	Pc%	F	P value	Pc%
Fly ash wt.% (A)	2	1183	0.001	61.61	283	0.004	55.11
Mg wt.% (B)	2	601	0.002	31.30	201	0.005	39.14
Impeller model (C)	2	133	0.007	6.92	28	0.034	5.45
Error	2			0.15			0.30

Dof - Degree of freedom; Pc-Percentage of contribution

Results of S/N Ratio:

The S/N ratio for each parameter level is determined by averaging the S/N ratios at the corresponding level. Process parameters with the highest S/N ratio would give the optimum quality with minimum variance. From the table.2 and response diagrams of S/N ratio (Fig. 2 and Fig. 3), it was found that the optimum parameters were fly ash wt.% (20% for hardness and 15wt.% for tensile strength), Mg wt.% (1.5) and impeller model (Radial flow).

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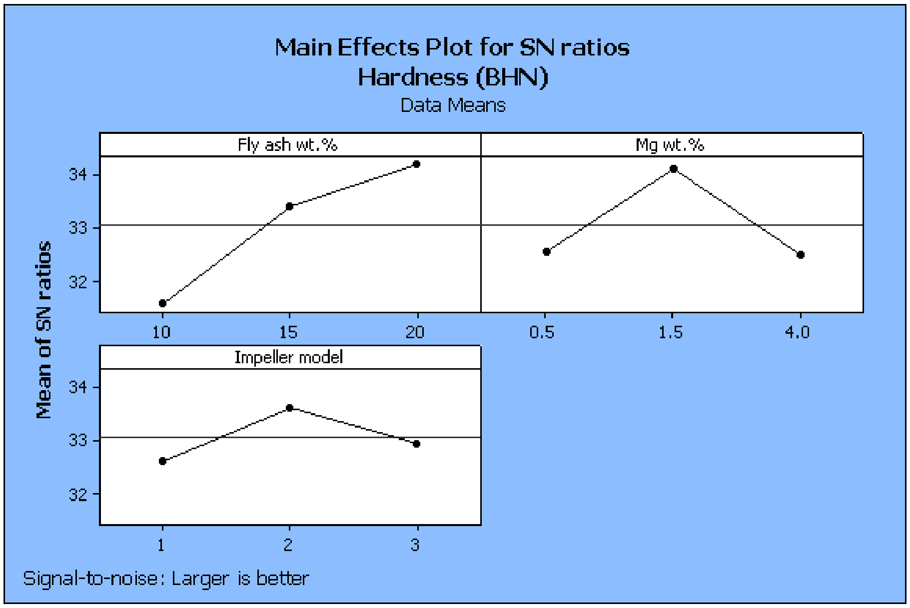

Fig. 2: Main Effects plot for SN ratios – Hardness.

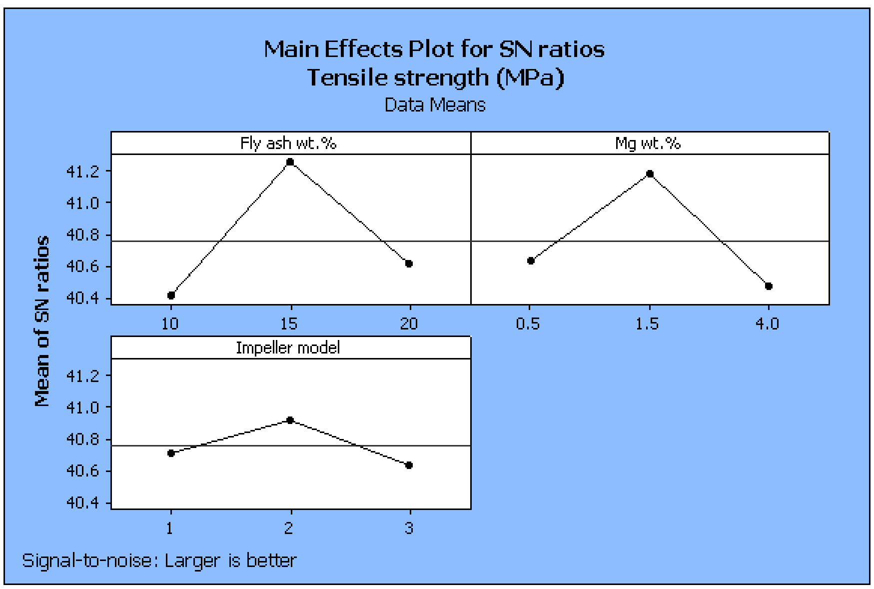

Fig. 3: Main Effects plot for SN ratios Tensile –Strength.

Results of ANOVA:

ANOVA determines the optimum combination of process parameters more accurately by investigating the relative importance among the parameters. ANOVA was performed with the help of the software package MINITAB15 for a level of significance of 5% to study the contribution of the factors. In the ANOVA table, there is a P-value for each independent parameter in the model. When the P-value is less than 0.05, then the parameter can be considered as statistically highly significant. It is observed that, all the three factors have less than 0.05, which means that they are highly significant at 95% confidence level. The last column of the table 3 shows the percentage contribution (Pc %) of each variable in the total variation indicating their degree of influence on the mechanical properties. It can be observed that the fly ash wt. % (61.61%) was the major contributing factor followed by Mg wt. % (31.30%) and finally impeller model (6.92%) influencing the hardness of the composites. The same trend was observed for the tensile strength of the composites. From the outcome of S/N ratio and ANOVA, it can be inferred that the results are closely matches with each other.

Multiple Linear Regression Models:

A multiple linear regression equation was developed to establish the correlation among the significant factors on the response. The value of regression coefficient, R^2 (0.9995) is in good agreement with the adjusted R^2 (0.9979) for hardness and R^2 (0.9981) is in good agreement with the adjusted R^2 (0.9922) for tensile strength. Since both the values are reasonably close to unity, models provide reasonably good explanation of the relationship between the independent factors and the response (properties).
The regression equation developed for hardness is
Hardness = 26.2 + 1.30 (A) - 0.72 (B) + 0.67 (C) Eq. 1
The regression equation developed for tensile strength is
Tensile strength = 109 + 0.233(A) - 1.15(B) - 0.33(C) Eq. 2

From the Eqn. (1) and Eqn. (2), it is observed that the fly ash wt. % (A) plays a major role on hardness and tensile strength followed by Mg wt. % (B) and impeller model (C).

Confirmation Test:

A confirmation test is the final step in the design of experiment process. It was found that the optimum parameters were fly ash wt. % (20%), Mg wt.% (1.5), and impeller model (Radial flow) for the hardness whereas fly ash wt. % (15%), Mg wt.% (1.5) and impeller model (Radial flow) for the tensile strength of composites. The confirmation experiments were conducted for the optimum parameters. Best results (maximum hardness 55 BHN and tensile strength 126 MPa) were found and comparison was made with computed values developed from the regression model. Experimental values and calculated values for hardness and tensile strength from the regression equation are nearly same with least error (± 6%). The resulting equations seem to be capable of predicting the mechanical properties to the acceptable level of accuracy. However if number of observations of performance characteristics are increased further these errors can be reduced.

Discussions:

Role of Magnesium:

As seen from the ANOVA results, the optimum Mg content was found to be 1.5wt. %.The most important consideration in the production of metal matrix composites by stir casting route is the difficulty of wettability between the reinforcement and the matrix material. Since the surface tension of Mg (0.599N/m) is lower than Al (0.760 N/m), the addition of Mg reduces the surface tension of the molten Al. Moreover, addition of Mg facilitates the formation of solid solution reaction elements and increases the dynamic viscosity of composite slurry which reduces the floating velocity of fly ash particles.

It also reduces the solid–liquid interfacial energy by aiding the chemical reaction at the interface surface between the Al and fly ash particles and form various solid solution reaction elements as shown in equations (3 – 7).

$3Mg + 4\,Al_2O_3 \leftrightarrow 2Al + 3MgAl_2O_4$ Eq. 3
$SiO_2 + 2Mg \leftrightarrow 2MgO + Si$ Eq. 4
$2SiO_2 + 2Al + Mg \leftrightarrow MgAl_2O_4 + 2Si$ Eq. 5
$2Mg + Si \leftrightarrow Mg_2Si$ Eq. 6
$4Al\,(l) + 3C(s) \rightarrow Al_4C_3(s)$ Eq. 7

Several researchers have reported that the strength of the Al matrix ceramic reinforcement composite is dependent on the magnesium-silicide precipitates. Depending on the existing processing conditions, the excess Si would react with Mg to form Mg_2Si as shown in Equation (6). It was reported that Mg_2Si precipitates when 5wt. % Mg is added to Aluminium (Kobashi and Choh, 1993). It was observed that Mg_2Si precipitates in AMCs containing more than 2 wt. % Mg (Ahlatci et al., 2003). It was concluded that the Al_2O_3 particles are unstable in Al- Mg alloys and reacts with the Mg to form spinel $MgAl_2O_4$ on the surfaces of oxidized particles, resulting in the increase of interfacial bonding strength (Lloyd, 1994).

It was reported that the bonding was achieved for an Al-Mg alloy based composite through the formation of $MgAl_2O_4$ layer by reaction at the solid-liquid interface (Levi et al.1978). It was reported that the optimum addition of Mg for obtaining good distribution and maximum mechanical properties to be around 1wt. %. The addition of Mg lower than the optimum value results in the formation of agglomerates of reinforcement particles (Sukumaran, 1995). Hence the presence of Mg in Al matrix during composite production strengthens the matrix and scavenges the oxygen from the surface of the dispersoid, leading to an increase in the surface energy of the dispersoid.

It may be noted that the diffusion of Mg and its subsequent incorporation into the reinforcement at the Al-fly ash interface could also result in Mg depletion from the matrix, accounting for the reduction of the Mg_2Si particle sizes which lead to a decrease in solid solution strengthening. Hence results in declining the mechanical properties of the composites as well as an increase in porosity.

On the other hand, when the content of Mg is higher than 6%, above 650˚C, fly ash particles tend to react with aluminium forms brittle compounds like Al_3Mg_2 and Al_4C_3 which deteriorates the mechanical properties of composite and also the fly ash particles agglomerate in the Al matrix again. Moreover, higher Mg content (more than 3 wt.%) composites would reduce the corrosion resistance when it is subjected to high temperature service in corrosive environments (Varley ,1970) and leads to the large gas enclosures in the composite due to higher viscosity of the composite slurry. Hence, it is proved that the proper Mg addition (1.5 wt. %) is beneficial to homogeneous distribution of fly ash particles in the Al matrix which enhances the mechanical properties of composites.

Impeller Models:

Three type's 4 bladed impeller models as radial flow (Rushton type), radial flow and axial flow (blade angle 45˚) were designed and fabricated with 0.7 I_{OD}/C_{ID} ratio (Impeller outer dia to Crucible inner dia). The impeller consists of four blades which are joined together at 180˚ to each other along a vertical axis and blades are fixed to the hub. A birds-eye view of the impeller models are shown in Fig.4- 6.

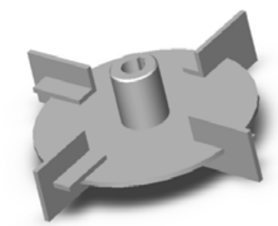

Fig. 4: Radial flow (Rushton type).

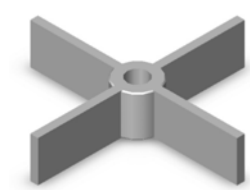

Fig. 5: Radial flow.

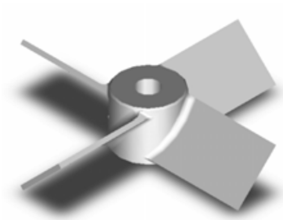

Fig. 6: Axial flow (45˚).

Axial flow impellers convey the composite slurry in the direction along the revolving axis (parallel to an axis of rotation of the impeller) and moves from the top to the bottom portion of the crucible as shown in Fig.7. It is preferred for low viscosity, high speed application like liquid-liquid dispersions. On the other hand the radial (Rushton type) flow impeller creates fluid flow radially outward from the impeller but mostly circulates into the region above the impeller and then slowly returns to the impeller zone by sedimentation. When compared with radial (Rushton type) flow impeller, the axial flow impeller produces a constant pumping action toward the bottom of the crucible followed by circulation to the top and a relatively rapid return to the impeller zone.

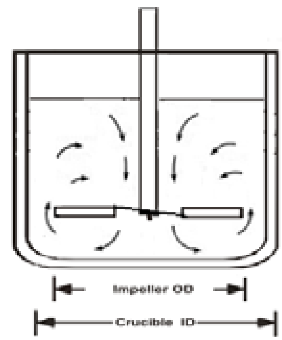

Fig. 7: Fluid flow pattern (axial flow impeller).

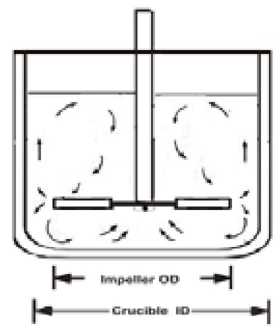

Fig. 8: Fluid flow pattern (radial flow impeller).

The radial flow impeller crates a radial flow pattern moving away from the impeller, towards the sides of the crucible as shown in Fig. 8. The flow impacts the side and moves in either an upward or downward direction to fill the top and bottom of the impeller.

From the response diagram of S/N ratio (Fig. 2 and Fig. 3), as would be expected, it was found that the 4 bladed radial flow impeller as the optimum model in obtaining the maximum mechanical properties of composite. Amongst axial and radial flow impellers, radial flow impeller which imposes essentially shear stress to the fluid could be used for mixing of high viscous fluids. It develops tangential flow with high shear rate which overcomes the cohesive force of the solid dendrites and disperses it into the individual particles, thus leading to a uniform microstructure. Shear force could also overcome the surface tension of the Al melt thereby improving the wettability between the molten Al and fly ash particles. The degree of homogenization depends on the rate of shear flow from the impeller to the clusters which are located away from its position. Therefore, rotating impeller must occupy a considerable proportion of the crucible to achieve the sufficient turbulence in the margin area. The effect of impeller model can be assessed only with respect to I_{OD}/C_{ID} ratio. It was observed that radial impeller with 0.7 I_{OD}/C_{ID} ratio ensures uniform distribution of fly ash particles in Al matrix and a little deposit of the slurry on the wall surface of the crucible.

Hence the impeller model with suitable I_{OD}/C_{ID} ratio has statistical and physical significance on the uniform distribution of fly ash particles in the composite. This was consequently reflected in the micro structure and mechanical properties acquired by the tensile and hardness tests carried out. Hence the shear force applied to the composite slurry by the axial flow and radial flow (Rushton type) impellers is not enough to disintegrate all the fly ash clusters. It can be concluded that impeller model (radial flow) and weight percentage of Mg (1.5 wt. %) have significant effect on distribution of the fly ash particle in the molten Al.

Micro structural Analysis:

Microstructures were examined on the Al -15wt % fly ash composite samples with a scanning electron microscope to reveal the particle distribution.

Fig.9 depicts SEM micrograph of sample containing Al-15 wt% fly ash developed by modified two-step mixing method of stir casting at the optimum parameters. High resolution micrograph shows the presence of clean interface between Al / fly ash for most of the fly ash particulates was almost perfectly bonded.

Fig. 9: Crucible view (Stirring with 0.7 I_{OD}/C_{ID} ratio).

Conclusions:

Based on this study, the following conclusions have been summarized.

The results of ANOVA revealed that the weight percentage of fly ash and Mg were the most significant parameters followed by impeller model. It was found that the optimum parameters were weight percentage of fly ash (15%) and weight percentage of Mg (1.5%), impeller model (Radial) for the tensile strength of composite materials.

The result of study suggests that the addition of fly ash content with Al increases the tensile strength and hardness of the composites compared to the Al. Addition of Mg up to 1.5% by weight into the composite slurry increases the wettability and thus increases the mechanical properties through strong interfacial bonding with fly ash particles. The verification experiment was conducted for the optimum parameters. The best results (maximum hardness 55 BHN and Tensile strength 126 MPa) have been obtained. The closeness of the results of predictions based on calculated S/N ratios and experimental values show that the Taguchi experimental technique can be used successfully for both optimization and prediction.

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